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# Mechanical properties of metal matrix composites based on TRIP steel and ZrO<sub>2</sub> ceramic foams

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#### Abstract

This study presents the mechanical behaviour of newly developed metal matrix composites (MMCs) in terms of compression and fatigue properties. The matrix of the MMCs consists of a high-alloyed metastable austenitic cast steel, which shows the TRIP-effect (TRIP-TRansformation Induced Plasticity). As reinforcing phase MgO partially stabilized  $ZrO_2$  is used which can also undergo a martensitic phase transformation. The samples were produced by infiltration of the cast steel into open foam structures made of  $ZrO_2$  with porosities of 30 and 50 ppi. The fatigue properties were investigated under total-strain and stress control. The martensitic phase transformation of the austen-itic steel matrix was investigated using a feritscope sensor.

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#### 1. Introduction

Metal-Matrix Composites (MMCs) have been investigated intensively in the last decades in order to combine the positive properties of the metallic and ceramic components. The resulting composite materials are characterized by an excellent mechanical strength, stiffness and wear resistance combined with a sufficient ductility [1]. The investigations were primarily focused on the reinforcement of light weight metals, e.g. aluminum, magnesium or titanium alloys. However, metal matrix composites based on a steel

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matrix have been examined rarely even though the resulting composite materials have a great potential in terms of strength and wear resistance.

The aim of the present study is the development of an innovative composite material based on highalloyed austenitic steels as matrix material and partially stabilized  $ZrO_2$  as reinforcing ceramic phase [2-3]. Both materials can show a martensitic phase transformation. Several research works have shown that the TRIP effect (Transformation Induced Plasticity) in the high-alloyed CrMnNi-steels results in a transformation of austenite into  $\varepsilon$ - and/or  $\alpha$ '-martensite which is accompanied by a significant improvement of strength and ductility [4-5]. Moreover, in former studies [6-7] it was shown that the TRIP-effect occurs in the non-reinforced cast steel also under cyclic loading influencing significantly the cyclic deformation behaviour.

The MgO partially stabilized  $ZrO_2$  can exist in three different modifications, i.e. cubic, tetragonal and monoclinic, respectively. A stress-induced transformation from the tetragonal phase into the monoclinic one increases the fracture toughness of materials based on partially stabilized zirconia. Moreover, the transformation results in a significant shear of the volume cell as well as in a volume increase generating compressive stresses [8-10] which can lead to a further strengthening of the MMCs.

In the collaborative research center "TRIP-Matrix Composite", two different manufacturing technologies are considered, i.e. powder metallurgy methods and melt infiltration of steel into ceramic preforms. This work presents first results about the deformation behaviour of MMCs produced by steel melt infiltration into zirconia open cell foams under compression and cyclic deformation.

#### 2. Experimental details

The investigated MMCs consist of an austenitic steel matrix with the nominal chemical composition of 16Cr - 6Mn - 6Ni (in wt.-%). This cast steel variant was already investigated intensively under static [4], dynamic [5] and cyclic deformation [6-7]. As ceramic preforms commercially available  $ZrO_2$ -filters, produced by Drache GmbH, Diez, Germany with porosities of 30 and 50 ppi (pores per inch) were used.



Fig. 1. (a) Schematic view of the cast system used for the production of the fatigue specimens. (b) Final shape of the fatigue specimens.

The MMCs for the compression tests were manufactured by the bottom-cast technique. The steel was melted in an induction furnace under argon atmosphere and was then infiltrated into the preheated ceramic preforms. The load direction lies vertical to the direction of infiltration.

The specimens for fatigue testing were produced with the cast system shown in Fig. 1 (a). The resulting specimens are partially reinforced with  $ZrO_2$ -filters within the gauge length. The casting mould is composed of an upper and lower part. The ashlar-shaped ceramic preforms (45 mm x 25 mm x 20 mm) were preheated to 1400°C to ensure a good infiltration. The infiltration occurs via a feeder head. The gripping part of the specimens is wedge-shaped in order to realize a directional solidification towards the feeder. The final shape was realized by precision turning, see Fig. 1 (b). The length and diameter of the gauge length was 20 mm and 15 mm, respectively. Thus, at least 10 cells were situated within the cross section of the gauge diameter. The complete specimen length was 164 mm.

Fig. 2 shows the initial microstructure of the infiltrated MMCs. The steel matrix is characterized by a dendritic cast microstructure with small amounts of  $\delta$ -ferrite. Fig. 2 indicates that the preforms are successfully infiltrated. The ceramic phase fractions of the 30 and 50 ppi ZrO<sub>2</sub>-filters amount 14% and 21%, respectively (determined using area analyses).

Compression tests were performed on a 200 kN servohydraulic testing system (MTS 810) using cylindrical specimens with a diameter and height of 9 mm. The strain rate was kept constant at  $4 \cdot 10^{-4}$  s<sup>-1</sup>.



Fig. 2. Optical micrographs of the infiltrated MMCs in the as-cast conditions, (a) 30 ppi and (b) 50 ppi.

Fatigue tests were performed at room temperature under total-strain or stress control, respectively, on a 250 kN servohydraulic testing system (MTS Landmark 250) using triangular load-time functions. A clipon extensometer with a gauge length of 10 mm was applied for strain measuring. As already mentioned, the TRIP effect in the steel matrix causes a transformation of the paramagnetic austenite into the ferromagnetic  $\alpha$ '-martensite. Thus, a feritscope (Helmut Fischer GmbH, Fischerscope MMS PC) was applied to follow the evolution of the ferromagnetic phase fraction during the fatigue tests.

#### 3. Results

#### 3.1. Compression properties

The technical compression stress-strain curves of the MMCs and unreinforced cast steel are plotted in Fig. 3 (a). The deformation behaviour of the MMCs can be described as ductile. The compression tests were interrupted at 50 % technical compressive strain without failure. The ceramic reinforcements cause an increase of the 0.2 % compressive yield strength which is more pronounced for the MMC with a porosity of 30 ppi (Fig. 3 (b)). At compressive strains higher than 6 %, the strength of the MMCs is lower than that of the cast steel indicating a continuous fracturing of the ceramic reinforcement. Consequently,

the compressive strength of the MMCs at 50 % strain is lower compared to the cast steel (Fig. 3 (b)). Figs. 3 (c) and (d) show the resulting deformation microstructures. As a result of the plastic deformation the austenitic steel matrix undergoes a transformation from austenite into  $\alpha$ <sup>c</sup>-martensite. A pronounced transformation can be observed in the vicinity of the steel-ceramic interface within characteristic deformation bands. The transformation occurs via an intermediate hexagonally indexed phase, called  $\varepsilon$ -martensite, as described elsewhere [5]. The ceramic reinforcement is broken into smaller particles causing an internal destruction of the composite material.



Fig. 3. (a) Compression stress-strain curve of the investigated MMCs compared to the cast steel 16Cr-6Mn-6Ni and (b) strength values. (c) + (d) Optical micrographs at different magnifications (colour etching) of the MMC (50 ppi) taken at a compressive strain of 50 %.

#### 3.2. Fatigue properties

#### (a) Total-strain controlled test

Some fatigue tests were carried out under symmetrical total-strain control ( $R_{\epsilon} = -1$ ) with strain amplitudes of  $\Delta \epsilon_t/2 = 2 \cdot 10^{-3}$  and  $3 \cdot 10^{-3}$  at a constant strain rate of  $4 \cdot 10^{-3}$  s<sup>-1</sup>. Fig. 4 (a) shows the stress response curves of the investigated MMCs compared to the unreinforced cast steel. The cyclic deformation behaviour of the cast steel at the investigated strain amplitudes is characterized by a small initial hardening, followed by a stage of cyclic softening and a pronounced secondary hardening, which is caused by an

ongoing martensitic transformation as described elsewhere [6]. Conversely, the MMCs show after a small initial hardening a continuous softening without a secondary hardening. Surface cracks could not be detected visually. However, the pronounced drop in the maximum stress is caused doubtless by the formation of crack networks, as marked in Fig. 5 (a) and (b) by arrows. The crack growth occurs preferentially at the steel-ceramic interface. Moreover, no pronounced  $\alpha$ '-martensite transformation was observed which is caused by the small amount of accumulated plastic strain. A local martensitic transformation was detected in the vicinity of localized stress concentration, e.g. short cracks, steel-ceramic interfaces and broken ceramic particles.



Fig. 4. (a) Cyclic stress response curves of the 50 ppi composite material compared to the unreinforced cast steel. (b) Evolution of the samples' stiffness under tension  $E_T$  and compression  $E_C$  obtained from the load reversals in the hysteresis loops.

Furthermore, the evolution of stiffness was determined using the slope of the unloadings after load reversals under tension ( $E_T$ ) and compression ( $E_C$ ). As shown in Fig. 4 (b), the stiffness of the cast steel is similar under compression and tension lying in the range of the elastic modulus of 191 GPa determined using an ultrasonic technique. Furthermore, in both cases a slight decrease of the stiffness with increasing number of cycles can be observed. At the end of the life time a strong decrease of  $E_T$  occurs whereas  $E_C$  remains nearly unaffected. This decrease in  $E_T$  is caused by an increasing long crack growth and crack opening under tensile stresses. Conversely, under compression the cracks are closed and can transfer the applied load without a significant loss in stiffness.



Fig. 5. Optical micrographs (colour etching) showing the deformation microstructure of the MMC with a 50 ppi foam. The specimen was deformed at  $\Delta \varepsilon_t/2 = 3 \cdot 10^{-3}$  and the micrograph was taken at N<sub>f</sub>. Load axis is vertical.

The elastic modulus of bulk  $ZrO_2$  is approximately 192 GPa [10]. However, the high porosity of the open cell ceramic foams results in an effective elastic modulus of approximately 100 – 110 GPa [11]. Assum-

ing the rule of mixture for a first approximation combined with a volume fraction of 20 %  $ZrO_2$ , the effective modulus of the composites should be approximately 170 GPa. The determined stiffness under compression is close to this calculated value. However, already in the first cycle  $E_T$  is much lower than  $E_C$  indicating that from the beginning of cyclic deformation internal damage occurs. The loss in stiffness under tension originates from rupture of ceramic particles or necks of the foam structure and/or from debonding of the ceramic phase from the steel matrix. Compared to the cast steel,  $E_T$  decreases much stronger with increasing number of cycles which is caused by an increasing number of failures within the material. The decrease in stress amplitude shown in Fig. 4 (a) is accompanied by a strong decrease of  $E_T$  whereas  $E_C$  remains constant.

(a) Stress-controlled multiple step test (MST)

Obviously, the most promising potential of the infiltrated MMCs will be found in structural parts which are subjected to compressive stresses. Therefore, some stress-controlled MSTs in the compressive regime were performed at a stress ratio of  $R_{\sigma} = -\infty$ . The stress amplitude of the initial level was 50 MPa. Every stress level was hold for  $10^3$  cycles and then the stress amplitude was increased by 50 MPa. Between every load level the stress amplitude was decreased for 100 cycles to the initial stress amplitude.



Fig. 6. Multiple step test (MST) at  $R_{\sigma} = -\infty$ . (a) Evolution of stress amplitude  $\Delta\sigma/2$  and mean strain  $\varepsilon_m$ . (b) Evolution of the strain amplitude  $\Delta\varepsilon_{t/2}$  and ferromagnetic phase fraction.

As shown in Fig. 6 (a), the cast steel is characterized by an increasing ratcheting behaviour with increasing stress amplitudes and thus an increasing negative mean strain, which is typical for samples subjected to mean stresses [12-13]. However, at all investigated stress amplitude levels a stabilization occurs caused by the  $\alpha$ '-martensite transformation and resulting cyclic hardening. The MMCs show at every stress amplitude level smaller mean strains compared to the unreinforced cast steel and thus a less pronounced cyclic ratcheting behaviour. Moreover, the mean strains of the MMC with a porosity of 30 ppi are lower than those of the MMC with a 50 ppi foam.

However, the resulting total strain amplitudes  $\Delta \epsilon_t/2$  of the MMCs are higher than those of the unreinforced cast steel (Fig. 6 (b)). Consequently, the MMCs show a larger plastic opening of the hysteresis loops indicating a more pronounced damage in the MMCs. Moreover, the MMC with the 30 ppi foam shows lower total strain amplitudes compared to the MMC with the 50 ppi ceramic foam. This is in agreement with the slightly higher 0.2 % compressive yield strength of the 30 ppi MMC. Thus, it can be

assumed that the higher amount of steel-ceramic interfaces results in an increasing number of crack initiation sites leading to larger plastic deformation.

Furthermore, the cast steel and the MMCs as well show in some cases a slightly decreasing strain amplitude within the load levels which is caused by the deformation induced  $\alpha^{\circ}$ -martensite transformation. Fig. 6 (b) shows exemplarily the evolution of the ferromagnetic phase fraction of the 30 ppi MMC. A significant martensite transformation in the MMCs was observed at stress amplitude levels higher than 150 MPa. The evolution is characterized by a plateau on every load level which is caused by the cyclic hardening and thus, decreasing plastic strain amplitude.

#### 4. Conclusions

The study of the monotonic and cyclic deformation behaviour of infiltrated MMCs leads to the following conclusions:

- (1) The ceramic reinforcement results in a slight increase of the 0.2 % compressive yield strength. The austenitic steel matrix is characterized by a martensitic transformation. The deformation behaviour of the composite specimens can be described as ductile.
- (2) Total-strain controlled fatigue tests have shown that tensile stresses and strains lead to a rapid generation of cracks at the steel-ceramic interface and within the ceramic necks. This damage results in a significant loss of material's stiffness under tension and a reduced fatigue life time. A pronounced martensitic transformation was not detected in agreement to a small accumulated plastic strain.
- (3) Stress-controlled multiple step tests in the compression regime revealed a less pronounced cyclic ratcheting of the MMCs compared to the cast steel. Nevertheless, the MMCs showed higher strain amplitudes indicating a more pronounced damage evolution.

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